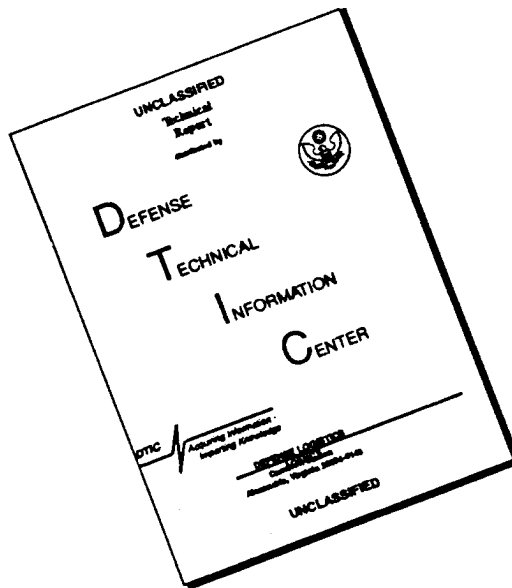


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Hydrothermal Destruction of Organics in Radford Army Ammunition Plant Water-Dry Waste Water: Bench-scale and Engineering-scale Testing

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March 1995

Prepared for Sam Houston State University
under Contract SHSU-5000-001



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19960912 080

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Summary and Conclusions

The main purpose of this work was to evaluate the reaction chemistry and required operating conditions, and obtain design-related data, for the hydrothermal destruction of organics in Radford Army's Ammunition Plant Water-Dry Waste Water (RAAP waste water). Another prime objective of this work was to demonstrate the use of a new pressure vessel, the Dual Shell Pressure Balanced Vessel (DSPBV), for processing such waste streams.

Bench-scale batch tests, bench-scale continuous tests, and a demonstration run with the engineering-scale DSPBV system were conducted. With the exception of some testing with the engineering-scale DSPBV system, H_2O_2 was added as the oxidant. During a portion of the demonstration run, air was introduced as the oxidant to the DSPBV reactor. The following conclusions and recommendations were drawn from the bench-scale and engineering-scale testing conducted on the RAAP waste water:

1. The waste water tested contained approximately 170 ppm ethanol, 63 ppm ether, and 65 ppm dinitrotoluene (DNT). The total organic carbon (TOC) and chemical oxygen demand (COD) concentrations were 150 ppm and 635 ppm, respectively.
2. Batch tests indicated that addition of 0.22 wt% H_2O_2 was sufficient to react with the TOC and COD present in the waste water. Bench-scale continuous tests indicated that even less H_2O_2 may be required (0.18 wt%).
3. The bench-scale and engineering-scale testing indicated that nitrates are being formed as the organics are being destroyed. This is most likely due to the destruction of DNT. The nitrate appears to be an intermediate product. If the operating temperature is sufficiently high (e.g., 365°C to 370°C) and residence times are 8 to 15 min (or longer), the nitrate levels drop. This drop is an indication that nitrate is reacting with organics in the waste, potentially converting the nitrate to nitrogen gas.
4. The bench-scale continuous data indicated that operation at approximately 370°C and a 15-min residence time, with addition of 0.18 wt% H_2O_2 , is sufficient to destroy approximately 98% of the TOC and COD.
5. The amount of off-gas produced/liter of liquid waste processed in the bench-scale continuous tests was 0.37 ± 0.08 L. The major gases detected in the off-gas were CO_2 , N_2 , and O_2 . The presence of O_2 in the off-gas was due to an excess amount of H_2O_2 that was added to the feed. The presence of nitrogen could be due to the breakdown of DNT to nitrate and then to nitrogen gas. More detailed off-gas analyses of selected runs showed that the only other carbon-containing gases detected were CO at a maximum level of 0.5% and methane at 0.013% (maximum). These analyses also showed that nitrous oxide or other nitrogen oxides were not detected in the off-gas (i.e., < 0.05%).

6. A nitrate destruction continuous bench-scale test, using ammonia to react with the nitrate, indicated that considerable nitrate destruction could be achieved at 365°C and a residence time of approximately 20 min. The nitrate appeared to react with the residual organics as well as with the ammonia.
7. In the engineering-scale DSPBV demonstration run, approximately 41 gal of waste water were processed at an average flow rate of approximately 0.42 L/min. The system operated well from a mechanical viewpoint. The only "mechanical" problem encountered was a leak in the air injection system. This leak is assumed to be the major cause for a lower-than-expected organic destruction efficiency when air was injected to the system.
8. The engineering-scale results were in good agreement with those obtained in the bench-scale continuous testing, indicating that the bench-scale data are applicable to the larger system.

The feed and product samples from the bench-scale and engineering-scale tests have been supplied to Sam Houston State University for organic speciation analyses, including DNT analyses. The results from these analyses will further refine the operating conditions that are required to destroy DNT in the RAAP waste water.

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1.0 Introduction

This report discusses work conducted by Battelle, Pacific Northwest Laboratories to evaluate the hydrothermal destruction of organics in Radford Army Ammunition Plant Water-Dry Waste Water (RAAP waste water). This waste water stream originates from the production of propellants. Ether and alcohol, which are used at certain stages in the single-base propellant manufacturing process, eventually must be removed to an acceptable level. First, the propellant is purged with heated nitrogen, primarily to remove the ether, and then it is treated with hot water to primarily remove the alcohol. During this water treatment phase, alcohol and some ether along with some dinitrotoluene (DNT) from the propellant enters into the water stream. The waste stream received from the water-dry operation, for this study, contained approximately 170 ppm ethanol, 63 ppm di-ethyl-ether, and 65 ppm DNT.

The hydrothermal testing included use of the NitRem process developed by Battelle (U.S. Pat. No. 5,221,486 June 22, 1993). This process incorporates wet-air oxidation with two significant advancements: 1) chemistry improvements to destroy nitrogen compounds (e.g., ammonia, nitrate, and organic-nitrogen) unaffected by traditional wet-air oxidation and 2) a new reactor design, the Dual Shell Pressure Balanced Vessel (DSPBV, U.S. Pat. No. 5,167,930 Dec. 1, 1992). These improvements enable a wider operating range and efficient use of materials of construction.

In NitRem/hydrothermal processing, the feed stream is pressurized to 3000 to 3500 psi and then heated to 350°C to 370°C (just below supercritical conditions for water). Air (or another oxidant such as H_2O_2) is injected into the stream, which is then fed into the reactor. Organics are oxidized and decomposed in the front end of the reactor. If nitrates, ammonia, and/or organo-nitrates are present in the waste stream, and require destruction, a reagent (standard, inexpensive) is injected into the reactor (further downstream) as needed.

The DSPBV was developed to address many corrosion problems encountered with conventional high-pressure vessels operating in the corrosive environments associated with hydrothermal destruction processes. For example, average corrosion rates from 23 to 80 mils/year in conventional reaction vessels have been reported by Rice et al. (1994) and Latanision and Shaw (1993). In addition, the corrosion is often uneven, and pitting may take place. These problems are overcome with the dual shell design.

The DSPBV uses conventional steels for the pressure barrier with a thin-walled alloy shell to contain the often corrosive, reacting fluid. The steel pressure barrier, and the thin-walled alloy shell are separated by a pressure-transfer fluid. The pressure between the pressure-transfer fluid and the reacting fluid is maintained by an external piston that is at room temperature. Sensors are located in the pressure-transfer fluid so that any breach of the inner shell can be detected. This design provides for safer operation and is less costly than conventional, all-alloy, high-pressure vessels. Innotek Corporation of Little Rock, Arkansas, is the licensee for the NitRem and DSPBV reactor technologies.

The DSPBV used in this study is sized to process 10 gal/hr of liquid feed at up to 400°C (750°F) and 5000 psia with a residence time of 10 min. The inner shell is constructed of Incoloy 825, and the pressure barrier wall is constructed of 1 1/4 Cr-1/2 Mo carbon steel.

The hydrothermal testing described in this report was divided into three tasks: bench-scale batch testing (Section 2.0); bench-scale continuous testing (Section 3.0); and an engineering-scale demonstration run (Section 4.0), utilizing the DSPBV developed by Battelle (see appendix). The bench-scale testing was conducted to determine favorable operating conditions for hydrothermal destruction, and to obtain reaction chemistry and kinetic-related information that can be used for operation and design of larger systems. The demonstration run was conducted to evaluate the operation of the DSPBV and to provide additional chemistry, kinetic-related data, and design-related data.

2.0 Bench-scale Batch Testing

2.1 Objectives

The batch tests were conducted to screen potential operating conditions that might be further examined in continuous bench-scale testing. The results from the batch tests were used to evaluate the overall chemistry involved in the destruction of the organics in the RAAP waste water stream. The RAAP waste water stream was also characterized as part of this task.

2.2 Test Approach

Three bench-scale batch tests were conducted in this study. For each test, a weighed quantity of RAAP waste water, approximately 300 g, was placed in a 1-L batch autoclave reactor. A predetermined amount of H_2O_2 (see Section 2.4) was then added to the waste water from a 30% H_2O_2 stock solution. The reactor was then sealed, purged with helium gas, and pressure tested at 1000 psig. Next, it was purged until approximately 100 psig helium was present, and then was vented through a wet test meter to determine the volume and moles of helium gas in the reactor. After the reactor was vented, it was repressurized to 100 psig, using helium as the cover gas. An initial gas sample was taken from the reactor at this time. The heater to the reactor was then turned on, and heating to the designated hold temperature was initiated.

The heatup time to the desired operating temperature took approximately 60 min in all of the tests conducted. The reactor was maintained at the desired operating temperature for another 60 min. During the testing, the reactor temperature and pressure were monitored and recorded.

At the completion of each run, the heater was turned off, and rapid cooling of the reactor contents was achieved by introducing cooling water through cooling coils inside the reactor. This method allowed the reactor contents to be cooled from approximately 360°C to 150°C in 10 min. After the reactor contents cooled, a second gas sample was taken, and the reactor was depressurized. The volume of gas was measured through a wet test meter as the depressurization took place. The reactor was then opened, and the liquid product was retrieved, weighed, and submitted for analyses.

2.3 Equipment Description

The 1-L batch autoclave reactor used for the experiments is illustrated in Figure 2.1. The reactor, manufactured by Autoclave Engineers, consists of standard heating and cooling systems and a stirring apparatus. It is also equipped with a stainless steel liner to contain the solution to be tested and facilitate cleanup between runs. The system is equipped with both liquid and gas sampling ports that can be operated remotely during tests, and the entire reactor is situated behind a 1/4-in. steel barricade with all controls and monitoring devices located outside of the barricade.

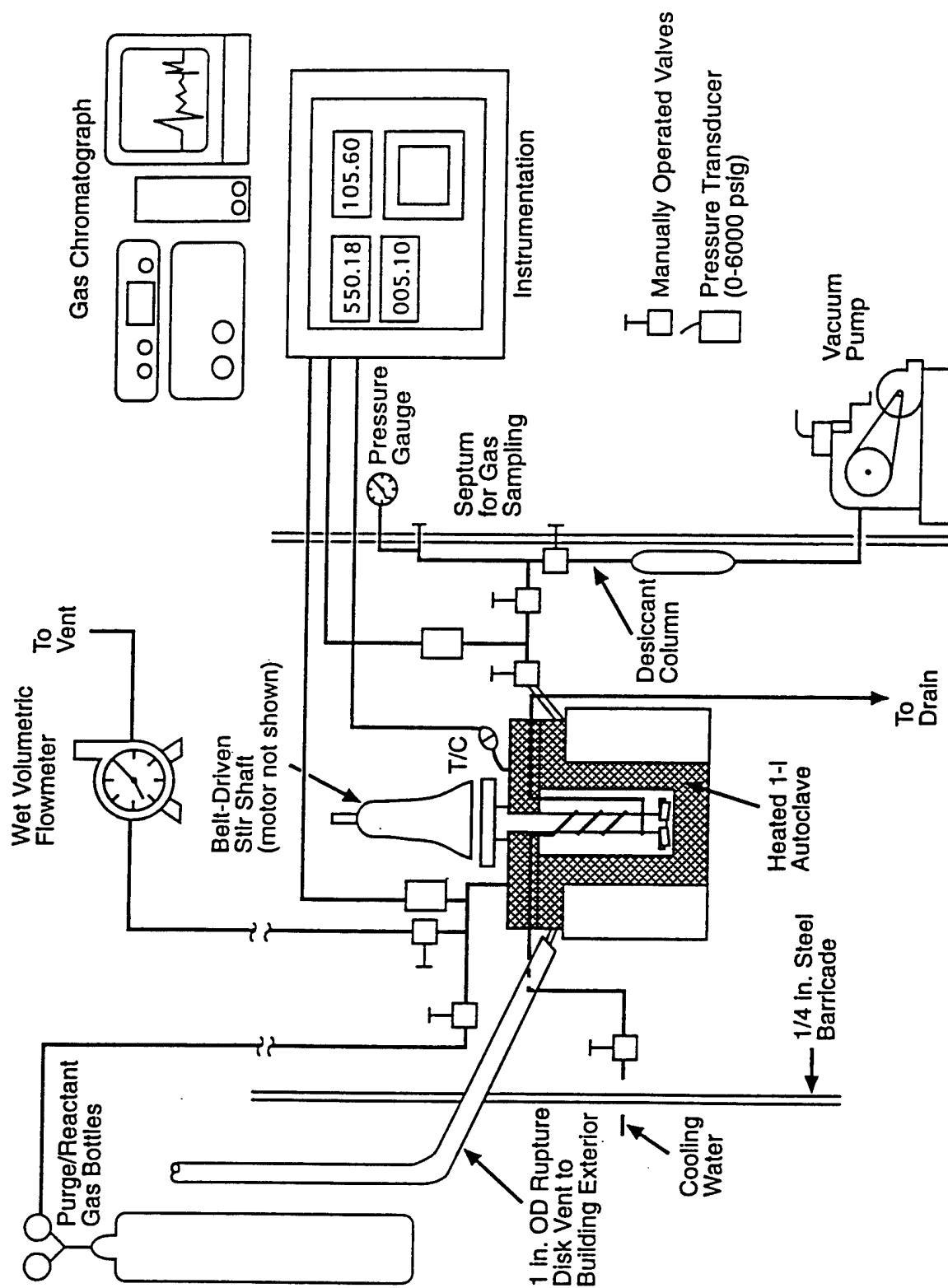


Figure 2.1. Schematic Diagram of the Bench-scale Batch Autoclave Reactor System

2.4 Test Conditions

For all three tests, H_2O_2 was added as the oxidant. The conditions of the tests are given in Table 2.1. The weight percent H_2O_2 that was added was estimated, based on the COD analyses, and on the assumption that 1 mole H_2O_2 resulted in 1 mole H_2O and 1/2 mole O_2 at the conditions tested. Based on this information, 0.22 wt% H_2O_2 represents approximately 50% excess oxidant.

Tests 1 and 2 were conducted primarily to evaluate the effect of temperature on the destruction of the organics in the feed stream. Test 3 was conducted to determine what effect, if any, the addition of more H_2O_2 had on the destruction of organics.

Table 2.1. Bench-scale Batch Test Conditions

Test	Operating Temperature (°C)	Operating Pressure (psig)	$\text{H}_2\text{O}_2^{(a)}$ in Feed (wt%)	Hold Time (minutes)
1	364	2729	0.22	60
2	347	2271	0.22	60
3	355	2527	0.44	60
(a) H_2O_2 was added as the oxidant to the RAAP waste water in these tests.				

2.5 Results and Discussion

The results of the feed stream analysis and batch testing are discussed below.

2.5.1 Feed Stream Characterization

The RAAP waste water stream was analyzed and characterized upon receipt. The chemical analyses are given in Table 2.2. The DNT analysis reported in Table 2.2 was supplied by Radford Army Ammunition Plant as their latest analysis of the stream. Feed and product samples from this testing have been supplied to Sam Houston State University for additional speciation, including DNT analyses.

Table 2.2. Feed Stream Analyses

Analyses Performed	Concentration
Chemical Oxygen Demand (ppm)	635
Total Organic Carbon (ppm)	150
Total Inorganic Carbon (ppm)	25
Nitrate (ppm)	0.5
Nitrite (ppm)	0.7
Ammonia (ppm)	<1
pH	6.1
Ethanol (ppm)	170
Di Ethyl Ether (ppm)	63
2,4 DNT (ppm) ^(a)	65
Ca ^(b)	20
K	6
Mg	7
Mn	1.4
Na	9
(a) Value reported by Radford Army Ammunition Plant as latest Water Dry-Waste Water Analysis.	
(b) Analysis showed other metals such as Fe, Zn, Cu, and Pb were present at <0.2 ppm.	

2.5.2 Batch Testing

The results from the batch tests are given in Table 2.3. For Test 1, the liquid effluent pH was comparable to the feed pH. In Test 2, the product pH was slightly lower than the feed pH; in Test 3, the product pH was significantly lower than the feed pH. The lower pH values could be a result of the formation of organic acid products or possibly HNO₃. In all cases, an increase in nitrate concentration was observed as compared to the feed stream. The highest nitrate levels were observed in Tests 2 and 3. The appearance of nitrate in the product streams is an indication that the organic nitrogen from DNT is being converted to nitrate. At the higher operating temperature (i.e., Test 1), the nitrate level

Table 2.3. Bench-scale Batch Test Results

Test	Liquid Effluent pH	Liquid Effluent Nitrate (ppm)	Liquid Effluent Nitrite (ppm)	Liquid Effluent Ammonia (ppm)	Liquid Effluent TIC ^(a) (ppm)
1	6.8	8.6	1.5	< 1	4.1
2	5.0	21.2	1.2	< 1	2.0
3	3.2	33.3	4.3	< 1	3.0
Test	Liquid Effluent TOC ^(b) (ppm)	Liquid Effluent COD ^(c) (ppm)	TOC destruction (%)	COD destruction (%)	% TOC as Acetate in Liquid Effluent
1	2.5	40	98.3	93.7	50
2	17	41	88.7	93.5	20
3	11	17	92.7	97.3	20
(a) TIC = total inorganic carbon. (b) TOC = total organic carbon. (c) COD = chemical oxygen demand.					

is substantially lower than in the other two runs, most likely a result of the nitrate reacting with residual organics at elevated temperatures. The oxidation of organics using nitrates or nitrites has been reported in previous studies under a variety of conditions (Cox et al. 1992; Orth et al. 1993; Schmidt et al. 1993; Foy et al. 1994). In all of the tests, the product nitrite levels were slightly higher, and the total inorganic carbon (TIC) levels were lower than those measured in the feed stream.

Chemical oxygen demand (COD) destruction ranged from approximately 94% to 97%. Total organic carbon (TOC) destruction ranged from 89% to 98%. In comparing the results, TOC destruction is considered a more accurate reading than COD because COD destruction is a colorimetric analytical technique, and color differences unrelated to COD destruction can thus affect the COD reading. This is especially true for the low-product COD levels being measured here. As would be expected, TOC destruction increased with increasing operating temperature. The product from Test 3 was visibly more yellow in color than in the other two tests. This color is most likely caused by increased corrosion of the stainless steel liner upon addition of more H₂O₂. The chromium concentration for the Test 3 product was 23 ppm versus approximately 0.3 ppm for Tests 1 and 2. This yellow color also most likely interferes with the colorimetric COD test, causing an "apparent" higher COD destruction level for Test 3 versus Tests 1 and 2.

Some organic speciation analyses were conducted to determine the source of the remaining TOC in the product. The samples were analyzed for oxalate, formate, and acetate. Of these organic species, only acetate was detected in the product samples. Between 20% and 50% of the remaining TOC is in

the form of acetate. The product samples from these tests have been provided to Sam Houston State University for further organics speciation, including DNT analyses.

In comparing the Test 2 and 3 results, it appears that the increase in H_2O_2 concentration had little, if any, effect on TOC destruction. The slight increase in TOC destruction in Test 3 as compared to Test 2 is most likely due to the slightly higher operating temperature.

Analyses of the off-gas from the batch testing showed that carbon dioxide was the only detectable carbon-containing gas in the off-gas. The off-gas also contained appreciable levels of oxygen and nitrogen. Some of the oxygen in the off-gas may be a result of unreacted oxygen from the degradation of H_2O_2 . The breakdown of DNT to nitrate and then conversion of some of the nitrate to nitrogen gas could be the source of the nitrogen in the off-gas.

3.0 Bench-scale Continuous Testing

3.1 Objectives

The bench-scale continuous tests were conducted to confirm promising operating conditions that were identified in the batch testing. Continuous testing provides chemistry and kinetic-related information that can be used for engineering-scale testing and for designing larger-scale systems.

3.2 Test Approach

Nine bench-scale continuous tests were conducted in this study. For each test, a measured quantity of RAAP waste water was placed in the feed tank. A predetermined amount of H_2O_2 (see Section 3.4) was then added to the waste water from a 30% H_2O_2 stock solution. At the beginning of a test, the heaters to the tubular reactor used for continuous testing were turned on. Water was introduced into the reactor at the target flow rate of the test to establish the specified run conditions (e.g., operating temperature, pressure, flow rate) under flow conditions. Target flow rates were predetermined so that the desired residence times in the reactor could be achieved (see Section 3.4). Once the desired operating conditions were established and maintained, the RAAP waste water was fed to the reactor.

During a run, feed line, reactor, and exit line pressures and temperatures were monitored and recorded. In addition, liquid feed and product flow rates and off-gas flow rates were monitored and recorded. Liquid product samples and off-gas samples were taken at approximately 1-hr intervals. These samples were then submitted for analyses.

3.3 Equipment Description

The continuous reactor system (CRS) used for the tests includes a 1-L 304 stainless steel tubular reactor, a feeding system, a product recovery system, and a data acquisition and control system (Figure 3.1). The dimensions of the 1-L reactor are 1 in. ID, 2 in. OD, and 72 in. length. In this configuration, the reactor is capable of operation at pressures up to 6000 psig and temperatures up to 450°C. Heat is supplied to the tubular reactor with a 6-kW, clamshell-style, three-zone furnace. The heating rate in each zone can be independently controlled. Feed is injected into the reactor with a high-pressure Milton Roy pump capable of pumping 0.25 to 3 L/hr at pressures from 1000 to 4500 psig.

The residence time of the feed in the reactor can be controlled by varying the pump feed rate, as well as by adjusting the effective reaction volume of the tubular reactor. The effective volume can be decreased with the addition of inert stainless steel packing as was done in all of these tests. With the turndown range of the pump, and using stainless steel packing, the reactor can be operated at residence times between 2 and 20 min.

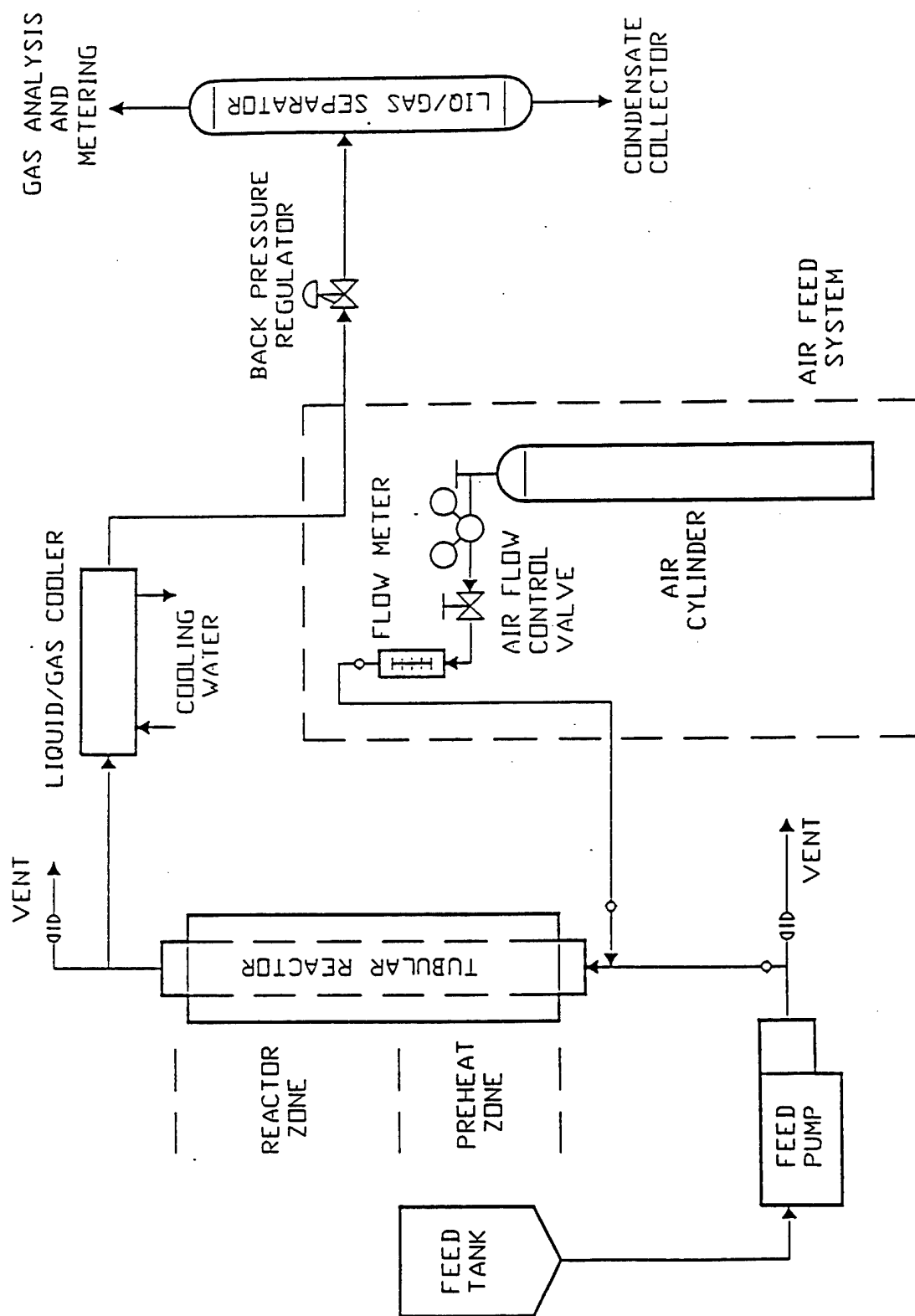


Figure 3.1. Schematic Diagram of the Bench-scale Continuous Reactor System

The reactor and the majority of the ancillary equipment under pressure are contained inside a 0.25-in. steel barricade. Temperature, pressure, feed rate indicators, controllers, and alarms are located outside the barricade.

3.4 Test Conditions

The conditions of the tests are given in Table 3.1. The tests have been arranged in Table 3.1 according to increasing temperature and residence time. Feed compositions were the same as those shown in Table 2.2, with the exception of Test 9. In the testing, three different operating temperatures were investigated: $342 \pm 3^\circ\text{C}$, $354 \pm 1^\circ\text{C}$, and $367 \pm 3^\circ\text{C}$. For each operating temperature, two different residence times were evaluated. In addition, Test 3 was conducted to determine whether less H_2O_2 could be added while achieving the same COD and TOC destruction. Test 8 was conducted as a repeat of Test 2 and to produce additional product for use in Test 9, which was conducted to evaluate the destruction of nitrate via reaction with ammonia.

Table 3.1. Bench-scale Continuous Test Conditions

Test	Operating Temperature ($^\circ\text{C}$)	Operating Pressure (psig)	H_2O_2 in Feed ^(a) (wt%)	Calculated Residence (minutes)
7	344	2942	0.18	8
6	341	2987	0.18	18
3	353	2999	0.17	9
1	354	2972	0.22	8
5	353	2978	0.18	16
2	368	3014	0.22	7
8	364	2984	0.21	8
4	370	3197	0.18	15
9 ^(b)	366	2979	—	18

(a) H_2O_2 was added as the oxidant to the RAAP waste water in these tests.
(b) Ammonium carbonate was added to the feed to react with and destroy the nitrate. The resulting ammonia concentration in the feed was 10.7 ppm.

3.5 Results and Discussion

The results from the continuous tests are provided in Table 3.2. The "organic destruction" tests (Tests 1 through 8) are discussed here, followed by the "nitrate destruction" test (Test 9).

3.5.1 Organic Destruction Tests

As shown in Table 3.2, the liquid product pH in the organic destruction tests ranged from 3.6 to 5.2. The liquid product nitrate levels ranged from 13 to 37 ppm. The highest nitrate concentrations were observed at the lower operating temperatures, while, for the most part, the lowest nitrate concentrations were observed at the highest operating temperatures. This is in fair agreement with what was observed in the batch studies, and is further evidence that the nitrate is being formed as an intermediate breakdown product from DNT destruction. At elevated temperatures (e.g., $> 365^{\circ}\text{C}$), the nitrate is most likely consumed by reaction with organics within the waste water stream. In all cases, less than 1 ppm nitrite and ammonia were detected in the product streams.

COD destruction ranged from 87.4% to 98.5%, and TOC destruction ranged from 81.3% to 97.7%. For each operating temperature investigated, COD and TOC destruction increased with increasing residence times. This is especially evident at the lower two operating temperatures. When comparing the results for the lower two operating temperatures, but equal approximate residence times, COD and TOC destructions are comparable, especially when comparing the 8 and 9 min data. However, when comparing the $367 \pm 3^{\circ}\text{C}$ with the lower temperature data and comparable residence times, the COD and TOC destruction at this higher operating temperature is always greater, as would be expected. Possible explanations may be 1) the initial organics may be broken down into more refractory organic compounds that require an elevated temperature (i.e., 365°C) to break them down further; 2) the organic compounds are breaking down into smaller organic fragments in the $354 \pm 1^{\circ}\text{C}$ runs, as compared to the $342 \pm 3^{\circ}\text{C}$, but these organics are not being broken down all the way to carbonate and, therefore, are still being measured as TOC. At $367 \pm 3^{\circ}\text{C}$, some of these smaller organic fragments are being broken down to carbonate. The product samples from these tests have been provided to Sam Houston State University for further organics speciation, including DNT analyses. These speciation analyses may provide additional insight into the two scenarios discussed above.

Significant amounts of the product TOC were present as acetate in all of the organic destruction tests, with the exception of Test 4. This test was conducted at the highest temperature and longest residence time, and only 6% of the TOC was present as acetate.

Comparable effluent stream concentrations resulted from Tests 1 and 3, indicating that addition of 0.17 wt% H_2O_2 is as effective as addition of 0.22 wt% H_2O_2 in treating the RAAP waste water stream.

In all of the bench-scale continuous organic destruction tests, the major gases detected in the off-gas were CO_2 , N_2 , and O_2 . The average off-gas concentrations were $24 \pm 3\%$ CO_2 , $31 \pm 6\%$ N_2 , and

Table 3.2. Bench-scale Continuous Test Results

Test	Liquid Effluent pH	Liquid Effluent Nitrate (ppm)	Liquid Effluent Nitrite (ppm)	Liquid Effluent Ammonia (ppm)	Liquid Effluent TIC ^(a) (ppm)
7	3.6	32	<1	<1	1
6	3.7	37	<1	<1	0.4
3	5.2	26	<1	---	8
1	4.5	15	<1	---	11
5	4.5	28	<1	<1	2
2	4.6	18	<1	---	6
8	4.7	19	<1	<1	1
4	4.8	13	<1	<1	2
g ^(d)	6.1	1.0	2.5	8.7	9
Test	Liquid Effluent TOC ^(b) (ppm)	Liquid Effluent COD ^(c) (ppm)	TOC destruction (%)	COD destruction (%)	% TOC as Acetate in Liquid Effluent
7	23	46	84.7	92.8	61
6	8.6	15	94.3	97.6	56
3	25	64	83.3	89.9	24
1	28	80	81.3	87.4	35
5	4.7	9.4	96.9	98.5	34
2	14	48	90.7	92.4	30
8	6.6	15	95.6	97.6	49
4	3.4	15	97.7	97.7	6
g ^(d)	2.4	---	86.7	---	100
<p>(a) TIC = total inorganic carbon. (b) TOC = total organic carbon. (c) COD = chemical oxygen demand. (d) The feed for this test was the combined product streams from Tests 2 and 8 + added ammonium carbonate; TOC = 10 ppm, TIC = 4 ppm, nitrite = 0.4 ppm, nitrate = 18 ppm, ammonia = 10.7 ppm, pH = 6.4.</p>					

$35 \pm 6\%$ O_2 . The presence of O_2 in the off-gas was due to an excess amount of H_2O_2 that was added to the feed. The presence of nitrogen could be due to the breakdown of DNT to nitrate and then to nitrogen gas. Off-gases from Tests 3 and 4 were submitted for more detailed gas analyses. The only other carbon-containing gases that were detected in these two analyses were CO at a maximum level of 0.5% (Test 4) and methane at 0.013% (Tests 4 and 5). The analyses of these two off-gas samples showed that nitrous oxide or other nitrogen oxides were not detected in the off-gas (i.e., $< 0.05\%$). Comparable amounts of off-gas/L of liquid feed were produced in all of the bench-scale continuous tests. An average of 0.37 ± 0.08 L off-gas were produced/L of liquid feed processed.

3.5.2 Nitrate Destruction Test

The TOC content and nitrate contents of the feed stream in Test 9 were 10 ppm and 18 ppm, respectively, and the pH was 6.4. Ammonia was then added as ammonium carbonate, resulting in an initial ammonia concentration of 10.7 ppm. Based on 18 ppm nitrate in the feed, and assuming 1.67 moles ammonia consume 1 mole of nitrate, 25% excess ammonia was determined to be present.

The Test 9 results, also given in Table 3.2, show that approximately 94% nitrate destruction and 77% TOC destruction were achieved. The ammonia level decreased slightly, but not as much as would be expected if it were reacting with all of the nitrate that was destroyed. The results indicate that some of the nitrate was being destroyed by reaction (i.e., oxidation) with some of the organics present in the feed as well as with the ammonia added to the feed. In any event, a significant reduction in the nitrate level was achieved. A small amount of nitrite was also measured in the effluent stream. Taking this nitrite into account, the total nitrate + nitrite (nitrogen) reduction was determined to be $> 81\%$ (assuming the initial nitrite concentration was 0 ppm).

4.0 Engineering-scale Testing

4.1 Objectives

The engineering-scale demonstration test was conducted to confirm that results from the bench-scale continuous testing were scalable to the larger scale. Also, the run was conducted to demonstrate the mechanical operation of the DSPBV pressure balancing concept. The data from the demonstration-scale run provide additional chemistry and kinetic-related information that can be used for design and operation of larger-scale systems.

4.2 Test Approach

The DSPBV was operated at Battelle for the engineering-scale demonstration. For the run, a measured quantity of RAAP waste water was pumped into the feed tank. As in the continuous testing, a predetermined amount of H_2O_2 (see Section 4.4) was then added to the waste water from a 30% H_2O_2 stock solution. At the beginning of the test, the heaters to the tubular reactor used in this system were turned on. Water was introduced into the reactor at the target flow rate of the test to establish the specified run conditions (e.g., operating temperature, pressure, flow rate). Once the desired operating conditions were established and maintained, the RAAP waste water was fed to the reactor.

At the completion of the scheduled run, using H_2O_2 as the oxidant, fresh feed (as-received RAAP waste water) was introduced to the feed tank. The feed was then routed to the reactor system. Air was introduced as the oxidant, and the demonstration run was continued.

During both phases of the run, feed line, reactor, and exit line pressures and temperatures were monitored and recorded. In addition, liquid feed and product flow rates and off-gas flow rates were monitored and recorded. Liquid product samples and off-gas samples were taken at approximately 1-hr intervals. These samples were then submitted for analyses.

4.3 Equipment Description

The DSPBV was developed to test and demonstrate a novel reactor design for moderate temperature, high-pressure, and corrosive conditions. The reactor and supporting equipment are described in more detail in the appendix, which was taken from Robertus et al. (1995). The DSPBV utilizes a thick steel outer shell as the pressure boundary and a thin disposable inner shell, which can be made from corrosion-resistant alloys that are appropriate for the waste to be treated. The outer and inner shells are separated by a pressure-transfer fluid. The pressure balance between the pressure-transfer fluid and the reacting fluid is maintained by an external piston that remains at ambient temperature. A second replaceable insert is used as a flow diverter so that reactants enter and products leave the same end of

the vessel. An internal resistance-style heating element is used to supply heat to the reacting mixture. Due to the modest upstream and downstream temperatures, ancillary equipment is made of stainless steel. This equipment includes a bank of six heat exchangers which recover heat from the products to preheat the feed stream. At the end of the process, a stainless steel autoclave is used as the pressure letdown device for separation of the gases and the effluent liquid. The system includes an air compressor so that air can be injected to the reactor.

The DSPBV is designed to process 10 gal/hr of dilute organic waste in water. Design working conditions are 370°C to 400°C at 3500 psig. Currently, the inner reactor shell, which is in contact with the waste water, is constructed of Incoloy 825, and the pressure barrier wall is constructed of 1 1/4 Cr-1/2 Mo carbon steel.

4.4 Operating Conditions

The operating conditions of the engineering-scale demonstration run are given in Table 4.1. In the scheduled demonstration run, H₂O₂ was added to the RAAP waste water as the oxidant (Test 1a in Table 4.1). Batch Tests 1 and 2 and Continuous Tests 1 through 3 provided the basis for selecting the amount of H₂O₂ to be added and for determining the operating conditions of the engineering-scale demonstration test. A H₂O₂ concentration of 0.18 wt%, an operating temperature of approximately 350°C, and a residence time of approximately 30 min were originally targeted. Upon evaluation of the data, the average operating temperature used was approximately 335°C, and the residence time was approximately 23 min.

Once the scheduled engineering-scale demonstration run was completed, as-received RAAP waste water was fed to the reactor, along with injection of air (Test 1b in Table 4.1), primarily to check out the air injection system. The target air flow rate had been calculated from the feed COD, and was determined to be 1.9 standard L/min (50% excess) based on a liquid feed flow rate of 0.5 L/min. An examination of the data indicated an air leak occurred upstream of the reactor, which translated to 0.6 standard L/min air being added to the reactor. This amount was less than the stoichiometric air requirement, based on the feed COD.

Table 4.1. Engineering-scale (DSPBV) Test Conditions

Test	Operating Temperature (°C)	Operating Pressure (psig)	H ₂ O ₂ in Feed ^(a) (wt%)	Calculated Residence Time (minutes)
1a	335	3250	0.18	23
1b	335	3250	0.6 standard liters/min ^(b)	18
(a) H ₂ O ₂ was added as the oxidant to the RAAP waste water in Test 1a.				
(b) Air was injected as the oxidant in Test 1b.				

Approximately 90 L (24 gal) of RAAP waste water were processed during Test 1a at an average flow rate of 0.44 L/min. An additional 65 L (17 gal) of RAAP waste water were processed during Test 1b. The average flow rate for this test was 0.41 L/min.

4.5 Results and Discussion

The engineering-scale equipment, including the pressure-balancing system, performed well from a mechanical standpoint. The air injection system also performed well with the exception of the leak upstream of the reactor.

The analyses of the liquid product streams from the engineering-scale test are given in Table 4.2. In all cases, the liquid product pH was lower than the feed pH of 6.1. The pH in Test 1a samples was slightly lower than Test 1b samples. In Test 1a, the liquid product nitrate level was approximately 15 ppm compared with 6.5 ppm (average) for Test 1b. In both cases, the nitrate level was greater than what was analyzed in the feed (0.5 ppm). These observations indicate that under both operating conditions DNT is being degraded, forming nitrate as one of the breakdown products. The observation of higher nitrate levels in Test 1a as compared with Test 1b may be an indication that more DNT is being degraded under the Test 1a conditions. Since these tests were conducted at approximately the same operating temperature and comparable residence times, it is unlikely that the nitrate is being consumed preferentially in Test 1b.

In all cases, liquid effluent levels of nitrite were less than 1 ppm, and TIC levels ranged from 7 to 16 ppm. The ammonia levels were low but measurable, (i.e., 5 to 7 ppm), in contrast to the batch and continuous tests in which no ammonia was detected (i.e., <1 ppm). In Test 1a, averages of 85% TOC and 93% COD destructions were achieved. In Test 1b, averages of only 72% TOC and 82.4% COD destructions were achieved. In both tests, the average %TOC as acetate were comparable; 29% and 24% for Test 1a and Test 1b, respectively.

The lower destruction levels that were obtained when using air instead of H_2O_2 is most likely due to the lower-than-expected air flow rate to the system. In addition, less efficient contacting of the air and the solution in the reactor may contribute somewhat to the lower destruction level when using air.

The results obtained for Test 1a agree well with those obtained in the bench-scale continuous tests. The Test 1a TOC and COD destruction levels are comparable to those obtained in the bench-scale runs that were conducted at $342 \pm 2^\circ C$ and $352 \pm 1^\circ C$ and 8 to 9 min residence times (see Table 3.1, Tests 1, 3, and 7). For example, at $344^\circ C$ and a residence time of 8 min, TOC destruction of 84.7% and COD destruction of 92.8% were achieved in Continuous Test 6. In comparison, at $335^\circ C$ and a residence time of 23 min, TOC destruction of 85.3% and COD destruction of 92.6% were achieved in Test 1a. This fairly good agreement indicates that the bench-scale continuous data are scalable and applicable to the engineering-scale system.

Table 4.2. Engineering-scale (DSPBV) Test Results

Test/time into test	Liquid Effluent pH	Liquid Effluent Nitrate (ppm)	Liquid Effluent Nitrite (ppm)	Liquid Effluent Ammonia(ppm)	Liquid Effluent TIC ^(a) (ppm)
1a/1 hr	5.5	14	<1	---	9
1a/2 hr	5.6	16	<1	5	9
1a/3 hr	5.5	15	<1	---	7
1a/3.3 hr	5.5	15	<1	5	13
1a/ average	5.5	15	<1	---	10
1b/1 hr	5.8	7.5	<1	---	11
1b/2 hr	5.8	5.5	<1	7	16
1b/ average	5.8	6.5	<1	---	14
Test/time into test	Liquid Effluent TOC ^(b) (ppm)	Liquid Effluent COD ^(c) (ppm)	TOC destruction (%)	COD ^(b) destruction (%)	% TOC as Acetate in Liquid Effluent
1a/1 hr	17	42	88.7	93.4	15
1a/2 hr	23	48	84.7	92.4	33
1a/3 hr	24	48	84.0	92.4	32
1a/3.3 hr	22	48	85.3	92.4	36
1a/ average	22	47	85.3	92.6	29
1b/1 hr	41	111	72.7	82.5	26
1b/2 hr	43	112	71.3	82.4	22
1b/ average	42	112	72.0	82.4	24
(a) TIC = total inorganic carbon.					
(b) TOC = total organic carbon.					
(c) COD = chemical oxygen demand.					

The product samples from these tests have been provided to Sam Houston State University for further organics speciation, including DNT analyses. These analyses will provide additional information regarding the operating conditions required to specifically destroy DNT.

5.0 References

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Appendix

Description of the Dual Shell Pressure Balanced Vessel

Appendix

Description of the Dual-Pressure-Balanced Vessel (DSPBV)

The Dual Shell Pressure-Balanced Vessel (DSPBV) was developed to test and demonstrate a novel reactor design for moderate temperature, high-pressure, corrosive conditions. The DSPBV utilizes electrical heat and an oxidant to convert an aqueous organic feedstock into clean water and an exhaust gas composed of mainly nitrogen and carbon dioxide. Conversion occurs at moderate temperature (350°C-400°C) and high pressure (3500-4000 psi). The DSPBV is designed to process 10 gal/hr of dilute organic waste in water.

The initial objective of the test system was to prove that the DSPBV concept will work safely in corrosive environments. Further objectives are to determine potential waste destruction efficiencies for various aqueous organic feedstocks that might be encountered in Hanford waste streams. Municipal sewage sludge effluent treatment is another targeted feedstock. Feedstocks that may be tested vary from dilute organic chemical bearing water wastes containing from 100 to 100,000 ppm organic contaminants to sewage sludge effluents having about 5% dry solids. Each feedstock is evaluated on an individual basis for environmental and health hazards. Testing with aqueous organic waste feedstocks takes place in the Materials Reliability Center (MRC) at Pacific Northwest Laboratory. The results generated from the testing will be used for design of larger scale treatment facilities.

A.1 Test Facility Description

The MRC contains a 100 ft by 50 ft high bay with adjoining office space. The building is conditioned with evaporative coolers and forced-air electric heaters mounted on the roof. The high bay contains a crane with a 20-ton capacity and 30-ft hook height, with access to the entire high bay floor area. The available utilities are:

- Richland city water for process and cooling water
- 480 V, 208 V, and 120 V electrical power
- 100 psig compressed air
- Sewer connections to the Richland publicly owned treatment works (POTW).

The MRC facility contains a sprinkler-water fire control system connected to the city water main.

A.2 Test System Description

Figure A.1 is a construction print for the DSPBV. The DSPBV uses a separate inner shell that fits close to the wall of the outer shell of the pressure vessel made of 1 1/4 Cr -1/2 Mo carbon steel (SA182 F11). The inner shell is constructed of Incoloy 825 (SB423, 424, 425). A pressure-transfer fluid,

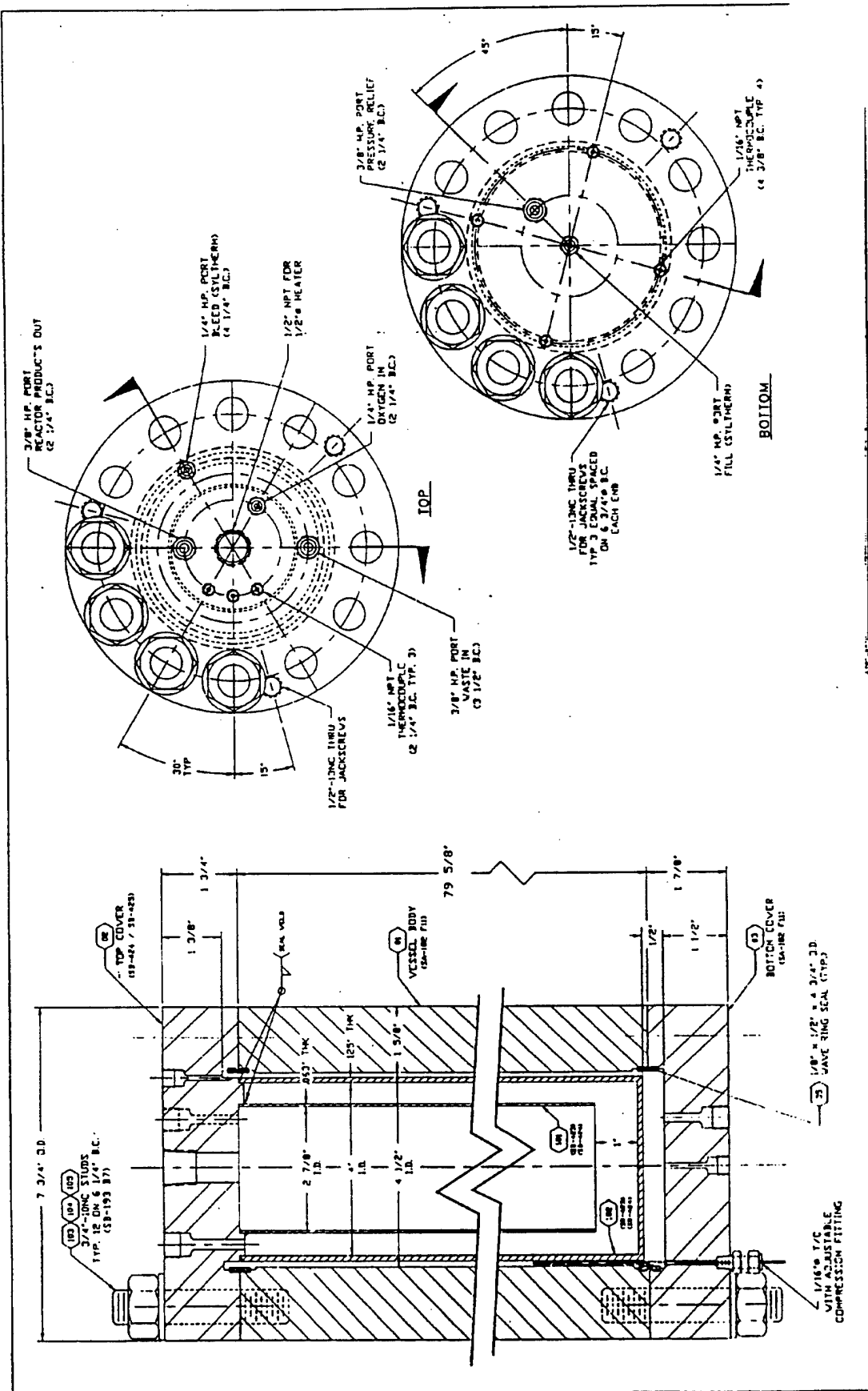


Figure A.1. DSR Construction Print

Syltherm 800, is placed in the annulus between the pressure vessel and the Incoloy 825 inner shell. The pressure-transfer fluid is kept physically separate and in hydrostatic equilibrium with the fluid entering the reactor. The pressure balancing is achieved by an external piston. Because the fluid pressure is balanced on both sides of the inner shell, the inner shell is not under significant stress; therefore, stress-corrosion cracking is minimized. The inner shell's thickness is determined by corrosion potential rather than strength; thus, it must only be thick enough to support its own weight and act as an envelope to separate the corrosive reactants from the carbon steel pressure vessel. The DSPBV design provides adequate space between the inner shell and the pressure vessel to allow for any degree of differential thermal expansion. The strength and corrosion resistance specifications of the pressure vessel are decoupled from the specifications of the inner shell.

While the pressure-transfer fluid balances the pressure, its electrical properties are continuously monitored to ensure the integrity of the inner shell. A significant change in conductivity of the pressure-transfer fluid alerts operators to a breach of the inner reactor so it can be replaced before the carbon steel pressure vessel corrodes. The ability to continuously monitor the integrity of the working reactor is a fundamental safety advance in reactor design.

The DSPBV working reactor unit is designed to monitor, protect, and preserve the integrity of the carbon steel pressure vessel. The working inner shell is designed for easy replacement as needed. The removable inner shell allows us to test alternate liner materials easily and inexpensively.

The DSPBV reactor is a test system designed for obtaining proof-of-principle information as well engineering data for the conversion of aqueous organic feedstocks into harmless gases and recyclable water. Capacity of the test system is 10 gal/hr liquid feed with a design flow rate of 10 gal/hr. Design working conditions are reactor temperatures of 700-750°F at 3500 psi. The DSPBV was built to ASME pressure vessel code for operation up to 750°F (399°C) and 5000 psia.

Figure A.2 shows a sketch of the fundamental pieces involved in the pressure balanced piston design. Other options studied but rejected for either time or money considerations included 1) a bellows, 2) a sliding o-ring seal, and 3) the inner shells with a stationary o-ring seal (instead of welds used in the final design).

The equipment illustrated in Figure A.2 can be grouped into six categories for purposes of discussion:

- the main reactor and pressure balancing piston
- process feed tanks, pump, and heat exchanger
- Syltherm supply tank
- high-pressure air supply system
- pressure letdown equipment
- instrumentation.

Figure A.3 shows schematically a picture of the main supporting equipment for the DSPBV. This includes the air supply system and the pressure letdown vessel.

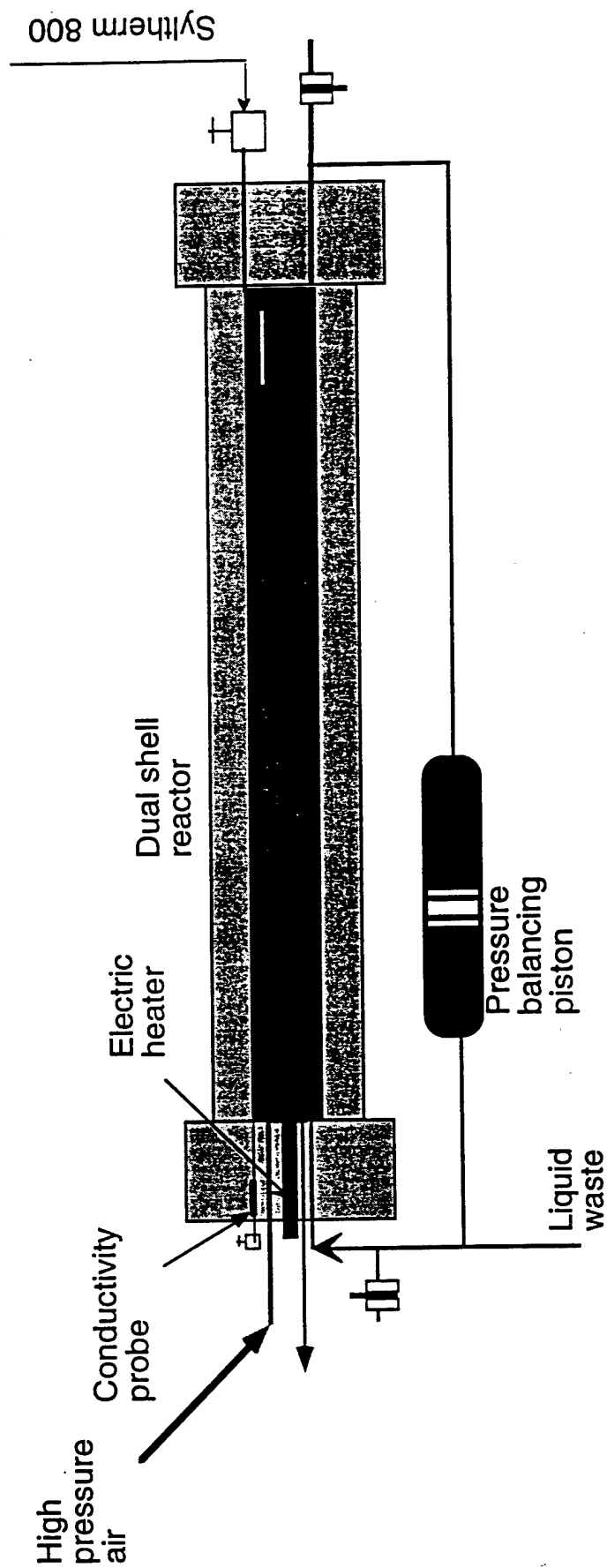


Figure A.2. Schematic of Reactor and Pressure-Balancing Piston

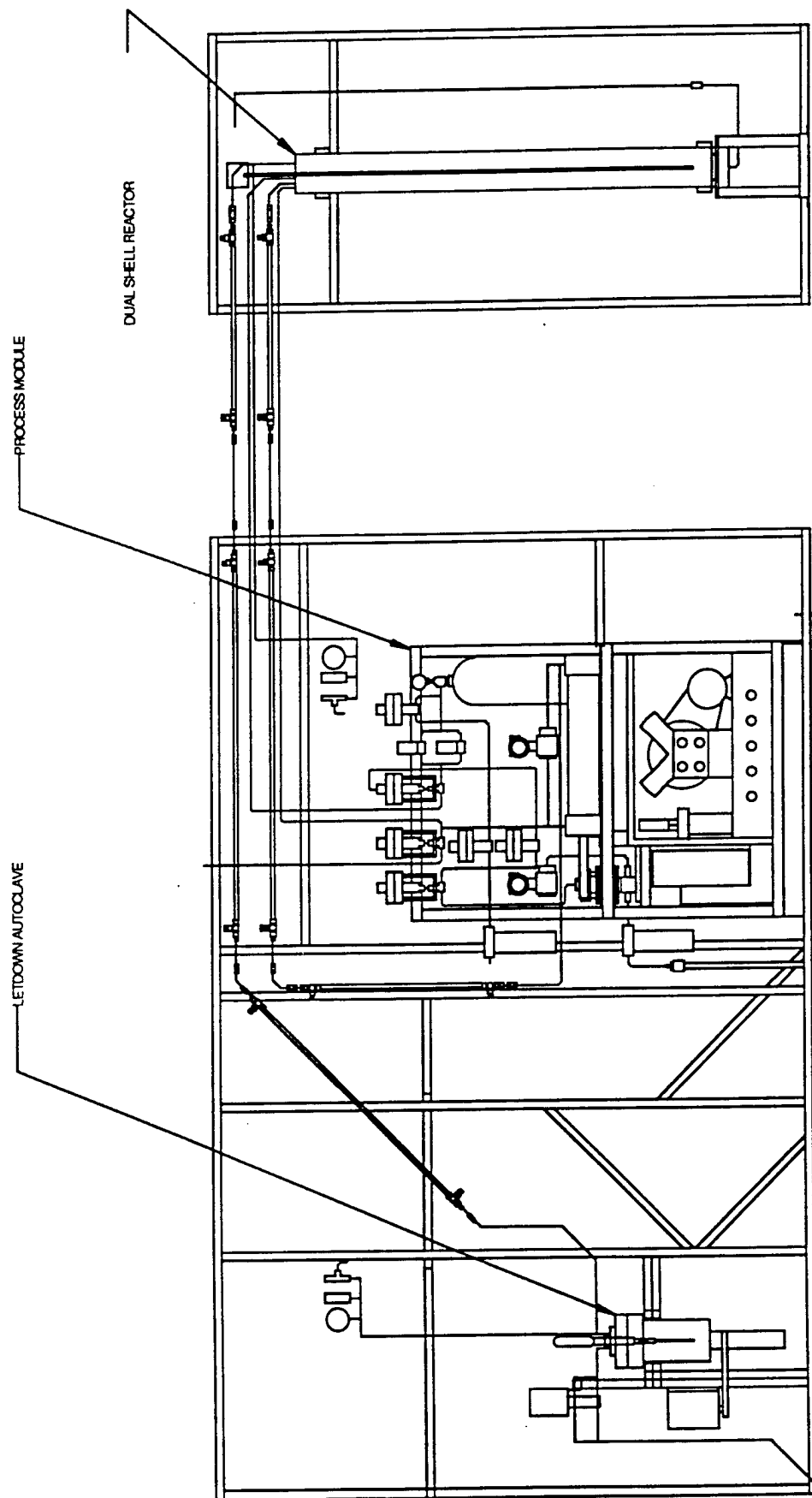


Figure A.3. DSR and Major Supporting Equipment

A.2.1 Process Feed Tanks and Pump

The process feed tank is polyethylene with a 50-gal capacity and has a metal lid to minimize evaporation into the room. The process feed tank is on an electronic scale which is tied to the data acquisition system. Weight readings are used to provide values for process feed flowrate to the reactor.

A second tank made of polyethylene contains deionized (DI) water for startup and for flushing out the reactor between runs. The flushing is done after each run to cool the reactor. A third tank is used to recirculate water through the shell of the product coolers and feed preheaters.

The process feed pump is an air operated, high-pressure, positive displacement pump. It will normally operate off building air and requires 28 SCFM at full capacity. The pump can be safely operated for at least 10 min using air from the storage cylinders of the high-pressure air system. This will allow safe shutdown of the system in the event of failure of the building air compressor. The tanks and pump are shown in Figure A.4.

A.2.2 Syltherm Supply Tank

The Syltherm supply tank is a carbon steel vessel protected with a safety relief valve that releases at 5 psig. The vessel is designed to have a nitrogen purge. When the reactor annulus is to be filled with Syltherm, a small pump whose suction line is in the Syltherm supply tank forces Syltherm into the bottom and out the top of the reactor. When the reactor annulus and pressure balancing piston are full, the fill valves at the top and bottom of the reactor are closed.

A.2.3 High-Pressure Air Supply System

The air supply system is an industrial unit typically used to fill self-contained breathing tanks for fire departments. The system can deliver up to 4 ACFM air at 6000 psig. The compressor is designed for intermittent use only. Consequently, two ASME vessels have been included to hold ~300 SCF (150 SCF each) air at 6000 psig. {Our operating storage tank pressure will be closer to 4000 psig}. Typical air consumption rates will be ~ 2 SCFM, so the demands on the compressor will not be too frequent once the tanks are full.

The complete air supply system is shown schematically in Figure A.5. The air compressor module including the two process air supply tanks has its own internal gauges and sensors to protect it from over-pressure, or over-temperature. It also cycles periodically (every 15 min) to remove liquid water that may have collected in the compressor. Outlet gas from the compressor to the air supply tanks is filtered to remove any entrained oil from the compressor.

A.2.4 Pressure Letdown Equipment

Product exiting the reactor passes through three heat exchangers. These heat exchangers share shell side fluid with another set of heat exchangers designed to preheat the incoming feed. Pressure letdown after cooling occurs in two stages. The first stage will drop the pressure from 5000 psia to 2500 psia across the main pressure regulating valve. This will release some dissolved gases (primarily CO₂). From there, the two-phase mixture will pass through a final heat exchanger and then expand into a 2-gal autoclave where liquid and gases will separate. Pressure on the autoclave is determined by the pressure set on the gas stream outlet. Liquid flow out the system is determined by the liquid level sensor.

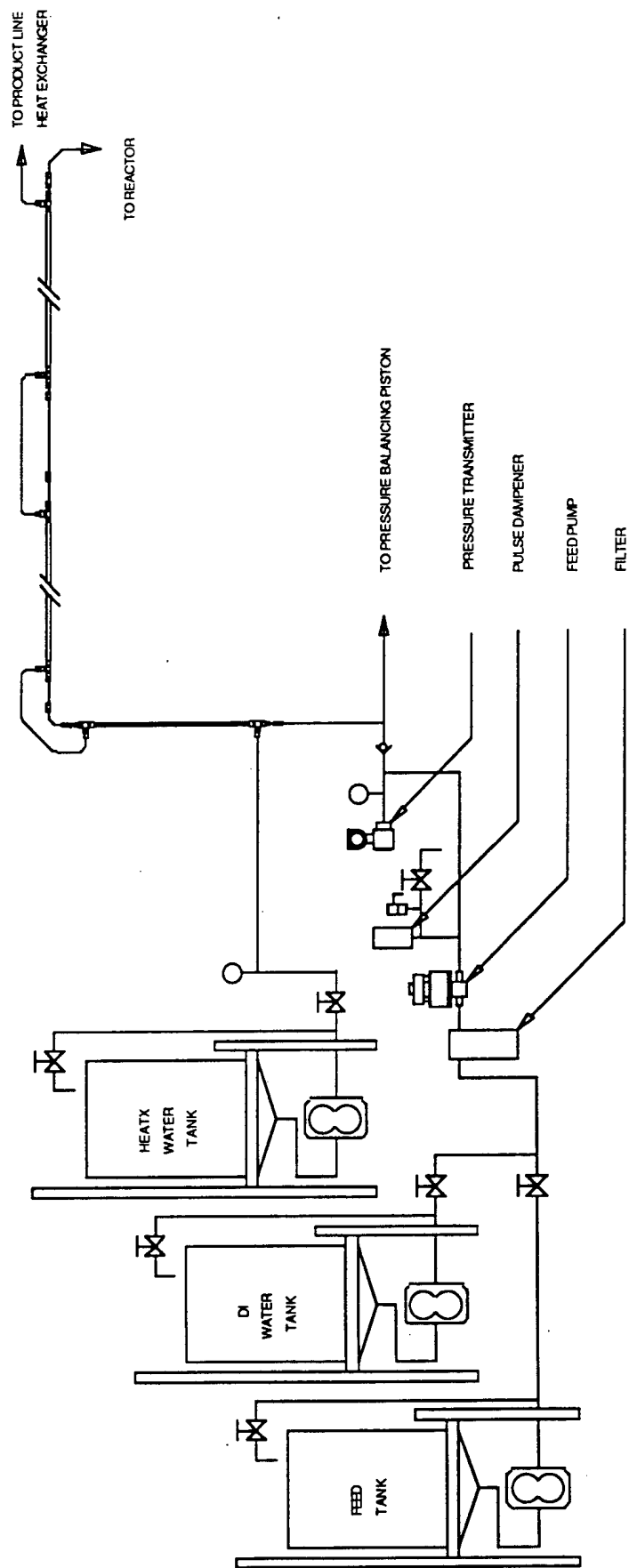


Figure A.4. Water and Feed Supply Tanks Plus Feed Pump

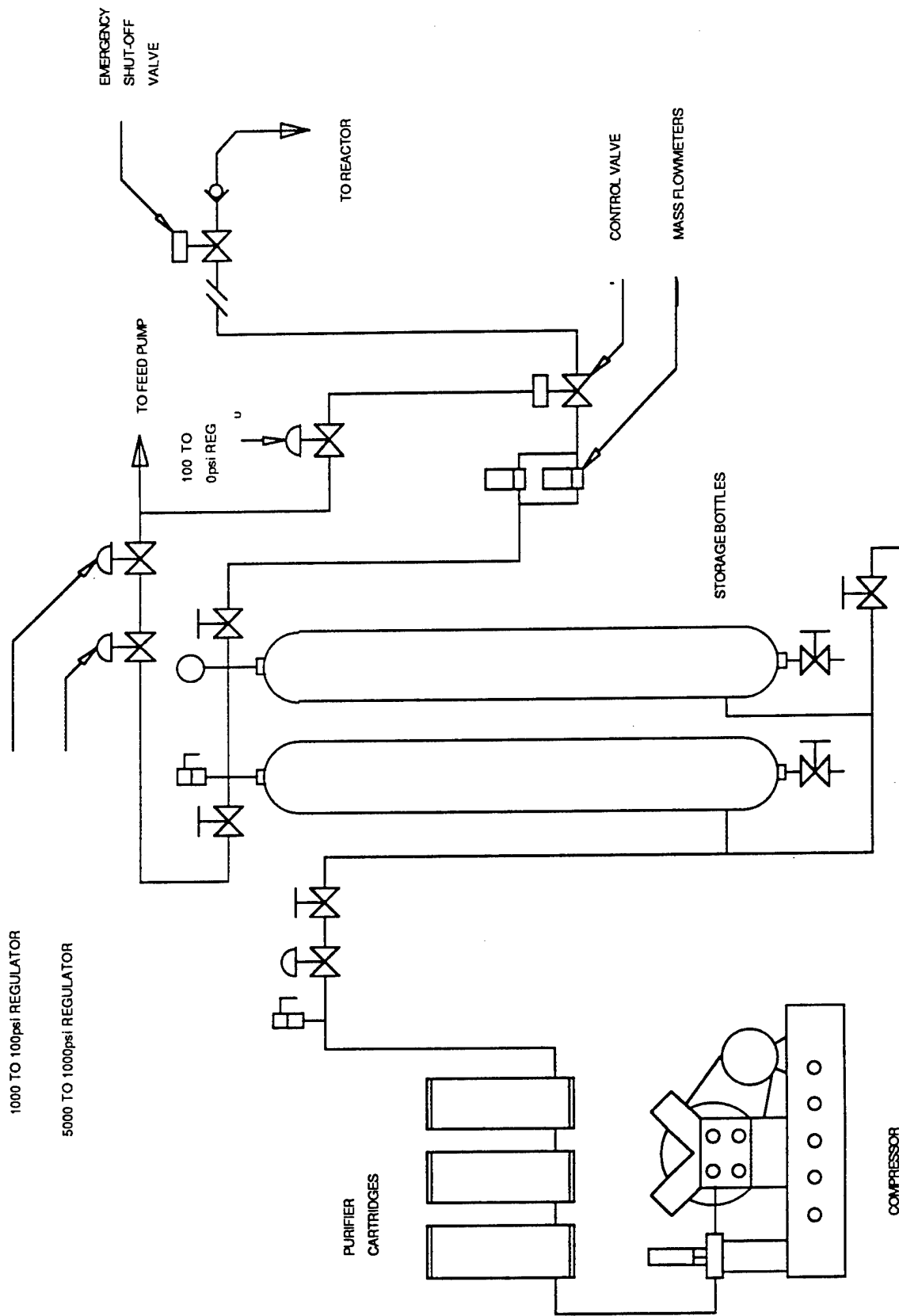


Figure A.5. Air Supply System for the DSPBV

A.2.5 Instrumentation

Type K thermocouples are used to monitor fluid temperatures into, inside, and out of the reactor. Pressure transducers monitor pressures. Electronic mass flow meters monitor air flow. Process liquid flow rate is determined by weight loss from the feed tank and by a turbine flow meter. The air supply is monitored continuously using a mass flow meter.

An FMC electropneumatic (NIT 200) controller sets the control parameters (proportional/integral) on the air supply to the liquid pump. Another control module monitors differential pressure across the pressure balancing piston. The controller will bleed off Syltherm if the Syltherm pressure exceeds the process feed pressure by more than 100 psi. It will also decrease the process feed flow rate if process liquid feed pressure exceeds the Syltherm pressure by more than 100 psi. Components of the control system are shown in Figure A.6.

For data reduction purposes there are two computer systems and one stripchart recorder collecting process data. The backup computer (IBM® clone) collects raw data (voltages or milliamps). The primary computer (Macintosh®) converts most signals to numbers meaningful for process control (temperatures/pressures/flows). The primary computer does no real-time process control but does provide operators with immediate feedback on process variables and sounds audible alarms when conditions are beyond specified limits.

Table A.1 summarizes the process variables being monitored and their positions at the recording locations. The temperature measurement locations are shown in Figure A.7. Traditional pressure gauges provide backup readings for the reactor fluids and the air supply cylinders.

Table A.1. Process Variables Measured and Recorded

Description	Abbr	T/C diist box	Recorder	IBM clone	Macintosh
Thermocouples					
Reactor liquid in @ 40"	RA40	1	1	1	1
Reactor liquid in @ 20 "	RA20	2	2	2	2
Reactor liquid in @ 0"	RA0	3	3	3	3
Reactor liquid out at 0"	RB0	4	4	4	4
Reactor liquid out @ 20"	RB20	5	5	5	5
Reactor liquid out @ 40"	RB40	6	6	6	6
Syltherm 800 @ 10"	RO10	7	7	7	7
Syltherm 800 @ 30"	RO30	8	8	8	8
Syltherm 800 @ 50"	RO50	9	9	9	17
Reactor skin @ 0"	RS0	10	10	10	18
Reactor skin @ 20"	RS20	11	11	11	19
Reactor skin @ 40 "	RS40	12	12	12	20
Heat exchanger 1 inlet	HX1A	13	13	13	21
Heat exchanger 2 inlet	HX2A	14	14	14	22
Heat exchanger 3 inlet	HX3A	15	15	15	23
Reactor inlet	RCTA	16	16	16	24
Heat exchanger 4 inlet	HX4A	17	17	17	25
Heat exchanger 5 inlet	HX5A	18	18	18	26
Heat exchanger 6 inlet	HX6A	19	19	19	27
Letdown vessel inlet	LDVA	21	20	20	28
Heat exchanger water inlet	HXWA	22	21	21	29
Heat exchanger water outlet	HXWB	20	22	22	30
spare		23			
spare		24			
Others					
Air flow "A"	MFMA		23	23	9
Air Flow "B"	MFMB		24	24	10
Reactor pressure	RCTP		25	25	11
Letdown vessel pressure	LDVP		26	26	12
Process feed line pressure	LIQP		27	27	13
DP Syltherm/process feed	OWDP		28	28	14
Feed Weight in Kg	WTKG		29	29	15
Feed flow in GPH	FGPH		30	30	16
spare			31	31	
spare			32	32	

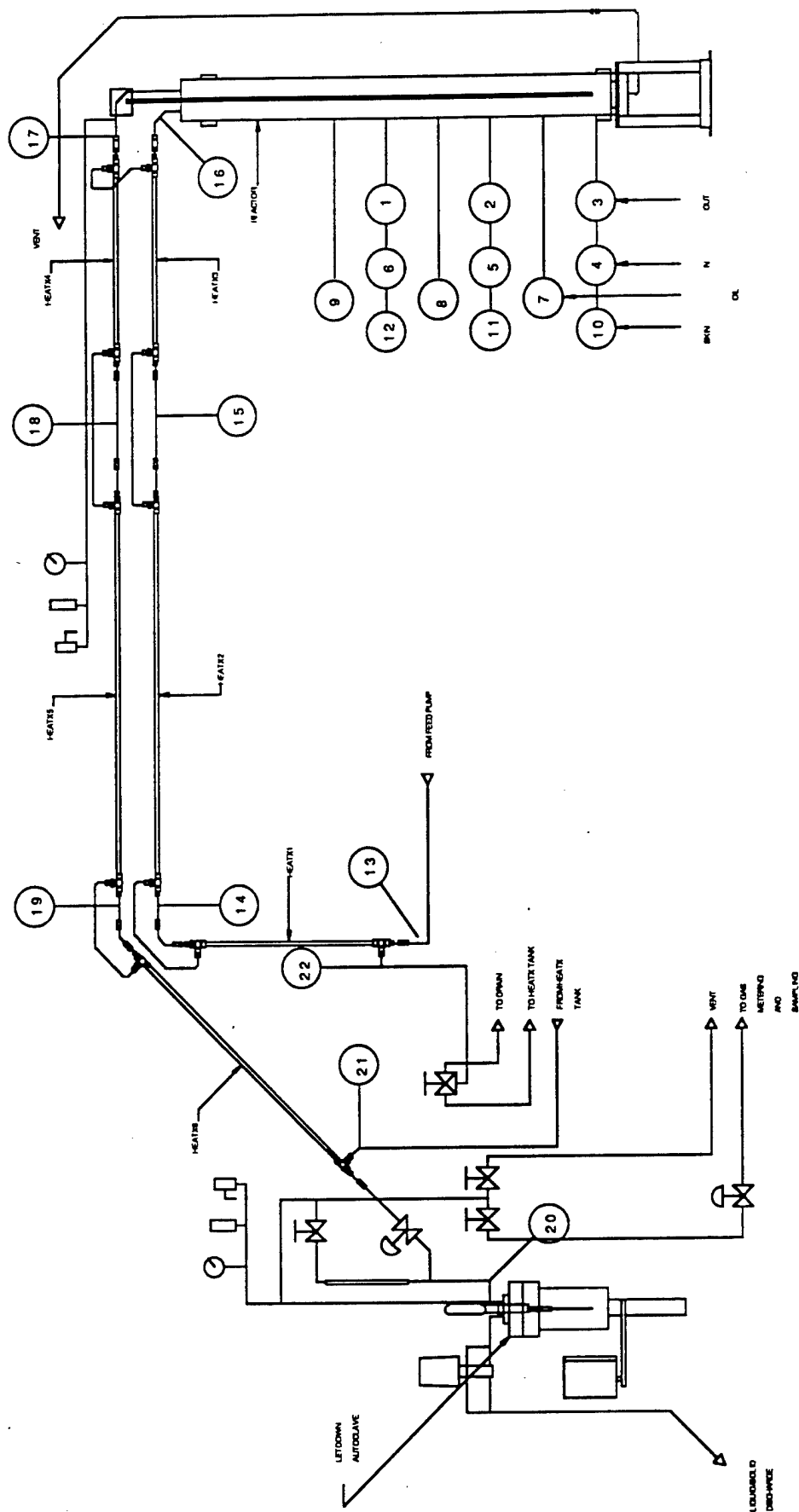


Figure A.7. Temperature Measurement Locations